

## FAT PRODUCING METHOD

### Technical Field

The present invention relates to a method for  
5 producing a fat.

### Background Art

Interesterification is one of effective means as a  
method for improving a fat. Conventionally,  
10 interesterification has been classified roughly into two  
methods, one is a chemical technique, that is, a metal  
catalytic method, wherein random interesterification is  
performed by using a substance such as an alkali metal  
alcoholate, an alkali metal, an alkali metal hydroxide, etc.  
15 as a catalyst, and the other is an enzymatic  
interesterification method, wherein of regiospecific or  
random interesterification is performed by using a lipase.

In the metal catalytic method and the enzymatic  
interesterification method, when the interesterification  
20 reaction is completed, an unreacted fatty acid ester  
remains, or diglycerides and monoglycerides or free fatty  
acids which are reaction by-products are produced. The  
presence of this fatty acid ester, diglycerides,  
monoglycerides and free fatty acids is known to have  
25 adverse effect on quality of an interesterified fat

occasionally. For example, in a method for producing high value added symmetric triglycerides, a typical thereof is cacao butter, obtained by enzymatic interesterification of triglycerides and a fatty acid ester using a lipase, since  
5 the aforementioned unnecessary components significantly influence on quality of high value added symmetric triglycerides, they should be removed as much as possible.

Usually, for producing 1,3-saturated-2-unsaturated triglycerides (hereinafter, sometimes, referred to as SUS),  
10 which are a major component of triglycerides, triglycerides (TG) and a fatty acid or its monohydric alcohol ester are subjected to an interesterification reaction with an enzyme. Since SUS triglycerides and fatty acids or their monohydric alcohol esters (FA) are produced, after the reaction, the  
15 fatty acids or their monohydric alcohol esters (FA) are distilled off by subjecting the reaction mixture to distillation-refining (purification). After distillation-refining, SUS triglycerides are concentrated by  
fractionation to increase a SUS component.

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#### Disclosure of the Invention

Then, under these circumstances, the present inventors aim at providing a method for suppressing isomerization as much as possible upon refining a fat by distillation.

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The present inventors have studied intensively and, as

the result, found that, upon distillation-refining of a reaction fat (reaction mixture) after completion of interesterification, when the reaction fat is refined by distillation under acidic conditions by addition of an  
5 organic acid, isomerization of a SUS component is suppressed. Thus, the present invention has been completed.

That is, the gist of the present invention is a method for producing a fat, which comprises removing a part or all of fatty acids or their monohydric alcohol esters (FA) from  
10 a mixture (MX) containing triglycerides (TG) and fatty acids or their monohydric alcohol esters (FA) by distillation-refining, wherein the removal is performed by adding an organic acid. The present invention includes the following features:

15 (1) The aforementioned method, wherein the mixture (MX) is a selective interesterification reaction product;

(2) The aforementioned method, wherein a total of triglycerides (TG) and fatty acids or their monohydric alcohol esters (FA) in the mixture (MX) is 95% or larger;

20 (3) The aforementioned method, wherein the organic acid is added by allowing to contact an aqueous organic acid solution with the mixture (MX);

(4) The aforementioned method, wherein after removal of only a part of fatty acids or their monohydric alcohol  
25 esters (FA) from the mixture (MX), a fresh fatty acid or

its monohydric alcohol ester (FA) is added, followed by  
subjecting the resulting mixture again to the selective  
interesterification reaction;

(5) The aforementioned method, wherein the removal of  
5 only a part of fatty acids or their monohydric alcohol  
esters (FA) from the mixture (MX) is performed at a  
temperature of at least 15°C lower than that at which the  
removal of all of fatty acids or their monohydric alcohol  
esters (FA) from the mixture (MX) is performed; and

10 (6) The aforementioned method, wherein the fresh  
fatty acid or its monohydric alcohol ester (FA) is a  
hydrogenated product of fatty acids or their monohydric  
alcohol esters (FA) separated from the mixture (MX).

#### 15 Best Mode for Performing the Invention

Examples of the aforementioned organic acid include  
citric acid, ascorbic acid, succinic acid, maleic acid, and  
oxalic acid. As a method for adding an organic acid to the  
mixture (MX), an organic acid can be added in the state  
20 that the organic acid is dissolved or dispersed in water or  
an aqueous solution containing a lower alcohol, or in the  
state of a powder. Preferably, the organic acid is added  
and dissolved in a reaction fat with mixing.

However, since solubility of an organic acid in the  
25 mixture (MX) is low and it is hardly soluble therein,

sometimes, the organic acid in a crystal state remains in the mixture (MX) and, when the mixture is subjected to distillation-refining as such, the organic acid is distilled off together with the fatty acids or their monohydric alcohol esters. Since such an organic acid thus distilled off tends to clog a piping of a distillation apparatus, preferably, an organic acid remaining in a crystal state in the reaction fat is removed by filtering or the like.

10       An amount of an organic acid to be added is not specifically limited, but an appropriate amount of an organic acid to be added is preferably 0.1 to 2% by weight relative to the mixture (MX). When the amount to be added is too small, isomerization suppressing effect is hardly  
15 obtainable. On the other hand, when an organic acid is excessively added, since the amount of an organic acid to be dissolved in the mixture (MX) is unchanged, isomerization suppressing effect is not increased. On the contrary, the aforementioned removal of an organic acid  
20 remaining in a crystal state without dissolved in the mixture (MX) is troublesome. Therefore, preferably, the aforementioned range of an organic acid is added.

      In the present invention, a typical example of the mixture (MX) is an interesterification reaction fat of  
25 triglycerides (TG) and fatty acids or their alcohol esters

(FA). This interesterification reaction is performed preferably in the presence of an enzyme having ester activity such as a lipase, more preferably in the presence of an immobilized or intracellular 1,3-regioselective  
5 lipase. Examples of the 1,3-regioselective lipase to be used for immobilization include lipases derived from microorganisms such as genus *Rhizopus*, genus *Aspergillus*, and genus *Mucor*, a pancreatic lipase, and the like.

While the mixture (MX) is not limited to a selective  
10 interesterification reaction fat, and the present invention is generally applicable to various fats and oils which are subjected to distillation-refining, the method of suppressing isomerization of the present invention greatly manifests isomerization suppressing effect on an  
15 interesterification reaction fat containing a SUS component, which can be used as a raw material fat of hard butter, as a main triglyceride component.

In case of an interesterification reaction fat containing a SUS component, which can be used as a raw  
20 material fat of hard butter, as a main triglyceride component, when an amount of unnecessary components (monoglycerides, diglycerides, etc.) is increased, functions as hard butter is reduced. Therefore, in the present invention, desirable, a total of triglycerides (TG)  
25 and fatty acids or their monohydric alcohol esters (FA) in

the mixture (MX) is 95% or larger, preferably 98% or larger.

In the present invention, when a fat having a high SUS component content is produced by interesterification of, in particular, ethyl stearate and a fat rich in oleic acid, it is advantageous to adopt a method comprising steps of: as a first step, subjecting ethyl stearate and a fat rich in oleic acid to interesterification, and distilling off a monohydric alcohol ester which binds to reacted oleic acid at the completion stage of the reaction; and then, as a second step, again subjecting a fresh ethyl stearate and the aforementioned interesterification reaction fat to interesterification to obtain an interesterification reaction fat having a SUS component content higher than that of the reaction fat of the first step, and distilling off a monohydric alcohol ester which binds to reacted oleic acid and monoglycerides or free fatty acids from such a reaction fat, to obtain an interesterification reaction fat having a high SUS component content. In the aforementioned method, a hydrogenated product of the monohydric alcohol ester which binds to reacted oleic acid and is distilled off at the first step distillation can be advantageously utilized in place of the fresh ethyl stearate to be used in the second step.

In the aforementioned method, the monohydric alcohol ester which binds to reacted oleic acid to be distilled off

at the first step distillation-refining can be distilled at a lower distillation temperature by distilling off only a part thereof. Then, heat history is small and an isomerization reaction can be relieved. In this case, preferably, the first step distillation is performed at a distillation temperature of at least 15°C lower than that at which the second step is performed.

When such a method is adopted, the first step distillation temperature for distilling off a monohydric alcohol ester which binds to reacted oleic acid after completion of the first step reaction is about 160°C to 200°C under reduced pressure (normally 5 torr or lower), and the second step distillation temperature for distilling off a monohydric alcohol ester which bind to reacted oleic acid and monoglycerides or free fatty acids after completion of the second step reaction is about 215 to 240°C under reduced pressure (normally 5 torr or lower). In the present invention, an organic acid is added and dissolved in reaction fats in both first step and second step distillation-refining. In this case, preferably, an insoluble acid is removed by filtering before distillation-refining. That is, when an organic acid remains, sometimes, a piping is clogged to cause troubles during distillation-refining. Therefore, preferably, such an organic acid should be removed.



## Examples

The following Examples illustrate embodiments of the present invention, but are merely illustration, and the spirit of the present invention is not restricted by them. In Examples, both parts and percents are by weight.

### Example 1

A mixture of 80 parts of commercially available ethyl stearate (C18 purity: 97.8%) and 20 parts of a high oleic sunflower oil was subjected to an interesterification reaction in a column packed with diatomaceous earth carrying a lipase having 1,3-specificity. After the reaction, an aqueous citric acid solution, in which citric acid had been dissolved in ion-exchanged water so that the final concentration became 0.2%, was added to the resulting reaction mixture, and the mixture was stirred at 80°C for 30 minutes. Then, the mixture was dehydrated at 110°C for 30 minutes, and precipitated citric acid was removed by filtration. The resulting mixture was subjected to distillation-refining (2 torr, 180°C, 60 minutes) to remove a part of fatty acids or their monohydric alcohol esters, and fat glycerides were concentrated. To the concentrated fat glycerides was added a mixture of the fatty acids or their monohydric alcohol esters distilled off the above distillation-refining, which had been extremely

hydrogenated until unsaturated fatty acid esters and unsaturated fatty acids became saturated fatty acid esters and saturated fatty acids, in such an amount that the triglyceride/ether ratio became 20/80. Then, a moisture content was adjusted to 70ppm, and the enzymatic interesterification reaction was performed again in a column. An aqueous citric acid solution was added again to the resulting reaction mixture of the latter interesterification, and the resulting mixture was subjected to distillation-refining (2 torr, 230°C, 90 minutes) to remove all of fatty acid esters and fatty acids, to obtain interesterified fat glycerides. The composition of the fat glycerides was analyzed by HPLC and TLC. In addition, 47 parts of the interesterified fat and 53 parts of a POP part fat ("Unilate P-110N" manufactured by Fuji Oil Co., Ltd.) were mixed and melted, and a cooling curve was measured based on a Jensen C.C. method (BS684: Section 1.13 Determination of cooling curve). The results of the analysis are shown in Table 1.

#### Example 2

According to the same manner as that described in Example 1, interesterified fat glycerides were obtained except that an aqueous ascorbic acid solution, in which ascorbic acid had been dissolved in ion-exchanged water so that the final concentration became 0.2%, was added to the

reaction mixture, the mixture was stirred at 80°C for 30 minutes and then dehydrated at 110°C for 30 minutes, precipitated ascorbic acid was removed by filtration, and then the resulting mixture was subjected to distillation-  
 5 refining. The results are shown in Table 1.

#### Comparative Example 1

According to the same manner as that described in Example 1, interesterified fat glycerides were obtained except that distillation-refining was performed without  
 10 treatment with citric acid. The results together with those of Examples 1 and 2 are shown in Table 1.

Table 1

	SOS	SSO	SSS	Jensen CC	
				T max	T min
Example 1	66.3	0.55	1.5	29.6	25.2
Example 2	66.1	0.58	1.6	29.6	25.1
Comparative Example 1	65.4	0.86	2.0	29.1	24.6

Further, chocolate was produced by using 12 parts of  
 15 the mixed fat (45 parts of the interesterified fat and 55 parts of POP part fat ("Unilate P-110N" manufactured by Fuji Oil Co., Ltd.)) obtained in each of Examples 1 and 2, and Comparative Example 1, 40 parts of cacao mass, 48 parts of powder sugar and 0.5 part of lecithin, and tempering  
 20 properties (peelability, broom resistance) were confirmed. As a result, although quality of Comparative Example 1 was good, quality of Examples 1 and 2 was extremely better as

compared with Comparative Example 1.

As a result of the foregoing, when an organic acid is added to a reaction fat upon distillation-refining after completion of interesterification, a SUS component is  
5 hardly isomerized, and an interesterified fat having better quality can be obtained.

#### Industrial Applicability

According to the present invention, there can be  
10 provided a method which can suppress isomerization of a SUS component which usually tends to occur during refining of a fat by distillation.